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DETERMINATION OF THE DISPERSION OF PORTLAND CEMENT THROUGHOUT A CONCRETE MIX BY NEUTRON ACTIVATION ANALYSIS

A THESIS

Presented to

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Approved:

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CHAPTER I

INTRODUCTION

One of the most important characteristics which control the quality of portland cement concrete is the uniformity of mixing. Adequately mixed, portland cement concrete exhibits such desirable qualities as optimum strength, workability, and durability. Because of the demand for concrete in building and road construction, it has become increasingly important to develop some simple method for the evaluation of mixing efficiency.

Manufacturers of concrete mixers are interested in evaluating the mixing efficiency of mixers having different shapes, different blade sizes, different blade angles, and rotating at various speeds. Producers of ready-mixed concrete are interested in securing a uniform mix in the minimum mixing time, and state highway departments, engineers, and other users of concrete are interested in obtaining concrete having optimum strength and durability from plants supplying concrete for buildings, bridges, and highways.

With a knowledge of the minimum time required for a specific mixer to produce concrete having a uniform dispersion of aggregate and cement throughout the mix, mixing time may well be reduced from the conventional time required by present specifications, and this reduction should result in increased capacity and lower production costs with no sacrifice in quality.

The use of radioactive isotopes in industrial processes and research is expanding rapidly. The fact that particle emissions from radioisotopes are easy to measure can simplify the identification of constituents of a sample. Irradiation in an accelerator or a reactor can make the sample radioactive, and a knowledge of the particle emissions from the sample then allows interpretation of the sample's composition. This thesis is the result of an investigation into the use of radioisotopes to determine the dispersion of portland cement throughout a concrete mix.

Various methods have been devised in the past for determining the cement content in a sample of concrete. The most prevalent method consists of determining the amount of soluble silica and calcium oxide in a sample by chemical analysis, and then indirectly calculating the percentage of cement by assuming some definite values of CaO and Si in the cement.[1] The method was devised for determining the cement content of a large sample of concrete, but can be used equally well in processing small mortar samples. This method is time-consuming, requires a well-equipped laboratory with trained personnel, and is not applicable to concrete containing aggregates such as slag, diatomites, and sodium silicates which liberate soluble silica under test conditions.

W.M. Dunagan suggests a test intended for use in the field for determining, prior to the initial hardening, the constituents of concrete.[2] According to this method, the sample is first weighed in

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[1] "Standard Method of Test for Cement Content of Hardened Portland Cement Concrete," Manual, American Society of Testing Materials, Part IV, 1958.

[2] W.M. Dunagan, "A Study of the Analysis of Fresh Concrete," Proceedings, A.S.T.M., Vol. 31, Part I, 1931, pp. 362-386.

air, then in water, and washed over a number 100 sieve. The aggregate is again weighed in water and the immersed weight of cement is obtained by the difference in the two submerged weights. It is necessary to know the specific gravity of cement to calculate its weight in air. An appreciable error enters the calculations in the assumption that all material passing the number 100 sieve is cement.

Another procedure for determining the cement content of a sample of freshly mixed concrete consists of using a heavy liquid and a centrifuge process for separating cement from the other ingredients of concrete.[3] The heavy media used comprises a liquid mixture of which the specific gravity may be adjusted to a value intermediate between that of cement and fine aggregate, thereby permitting the cement to sink and the aggregate to float. By means of appropriate calibration curves, cement content may be estimated.

The basis of a method by L.J. Murdock is the determination of the specific gravity of a cement suspension.[4] After washing a sample of fresh concrete over a number 100 sieve, hydrometer readings are recorded of the suspension collected. By reference to a control curve obtained from hydrometer readings of water in which known quantities of cement are suspended, the amount of cement can be determined. Here again, the assumption that all material passing through the number 100 sieve is cement creates an appreciable error in the calculations.

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[3] W.G. Hime and R.A. Willis, "A Method for the Determination of Cement Content of Plastic Concrete," Bulletin No. 209, A.S.T.M., Oct. 1955, pp. 37-43.

[4] L.J. Murdock, "The Determination of the Properties of Concrete," Cement and Lime Manufacture, Vol. 21, 1958, pp. 91-96.

Two additional methods for determining cement content were developed by L. R. Chadda.[5] In the first method, cement content is estimated by a conductimetric method based upon the determination of conductivity of pure water in which known quantities of unset cement-sand mixture have been shaken. From a standard curve showing the relationship between cement concentration and conductivity, the cement content of a sample can be interpolated from its conductivity measurement. Chadda's other method for determining cement content is based upon the differential absorption characteristics of cement and sand particles. The percent absorption increases as the concentration of cement increases in the mixture.

The latter two methods can be satisfactorily employed only for the determination of cement content in a freshly prepared cement-sand mixture to which no water has been added.

Previous research in this field has been primarily concerned with methods for spot checking samples of fresh concrete to insure a contractor's adherence to design specifications as to the amount of cement present. To the author's knowledge, no attempt has been made to determine the uniformity of cement dispersion throughout the concrete mix. The purpose of this research is to develop a method which will enable manufacturers of mixers, producers of ready-mixed concrete, and users of concrete to investigate the distribution of cement in a mixer operating under a given set of conditions, thereby allowing the optimum mixing time to be determined. In evaluating mixing efficiency, a sampling program

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[5] L. R. Chadda, "The Rapid Determination of Cement Content in Concrete and Mortar," Indian Concrete Journal, Vol. 29, No. 8, Aug. 1955, pp. 258-260.

and a rapid and accurate method for determining cement content of mortar samples will be developed.

CHAPTER II

EQUIPMENT FOR ACTIVATION ANALYSIS

Van de Graaff Accelerator.--Activation of cast mortar samples was accomplished through the use of Georgia Tech's one-million-volt Van de Graaff positive ion accelerator (made by the High Voltage Engineering Corporation).

The Van de Graaff is a special type of electrostatic accelerator which has a highly insulated terminal and a means of maintaining the terminal at a very high static potential with respect to ground. An ion injected into the high potential end of the machine is accelerated and directed downward through an evacuated acceleration tube to ground by the electrostatic field.

As an ion source, a mixture of ordinary and heavy hydrogen (deuterium) is used, which gives a beam containing about 25 micro amperes each of protons and deuterons. The 25 micro ampere mixed beam of deuterons and H_2^+ ions is directed at one million electron volts through the evacuated tube on a target of beryllium metal producing the reaction

$$ {}_4Be^9 + {}_1d^2 \rightarrow {}_5B^{10} + {}_0n^1 + \text{energy} $$

In this experiment the Van de Graaff was used for the production of neutrons for irradiation of the mortar samples.

A small general-purpose thermal neutron irradiator as shown in Figure 1 was constructed for use with the Van de Graaff in performing this project. The beryllium target is surrounded by a mass of paraffin

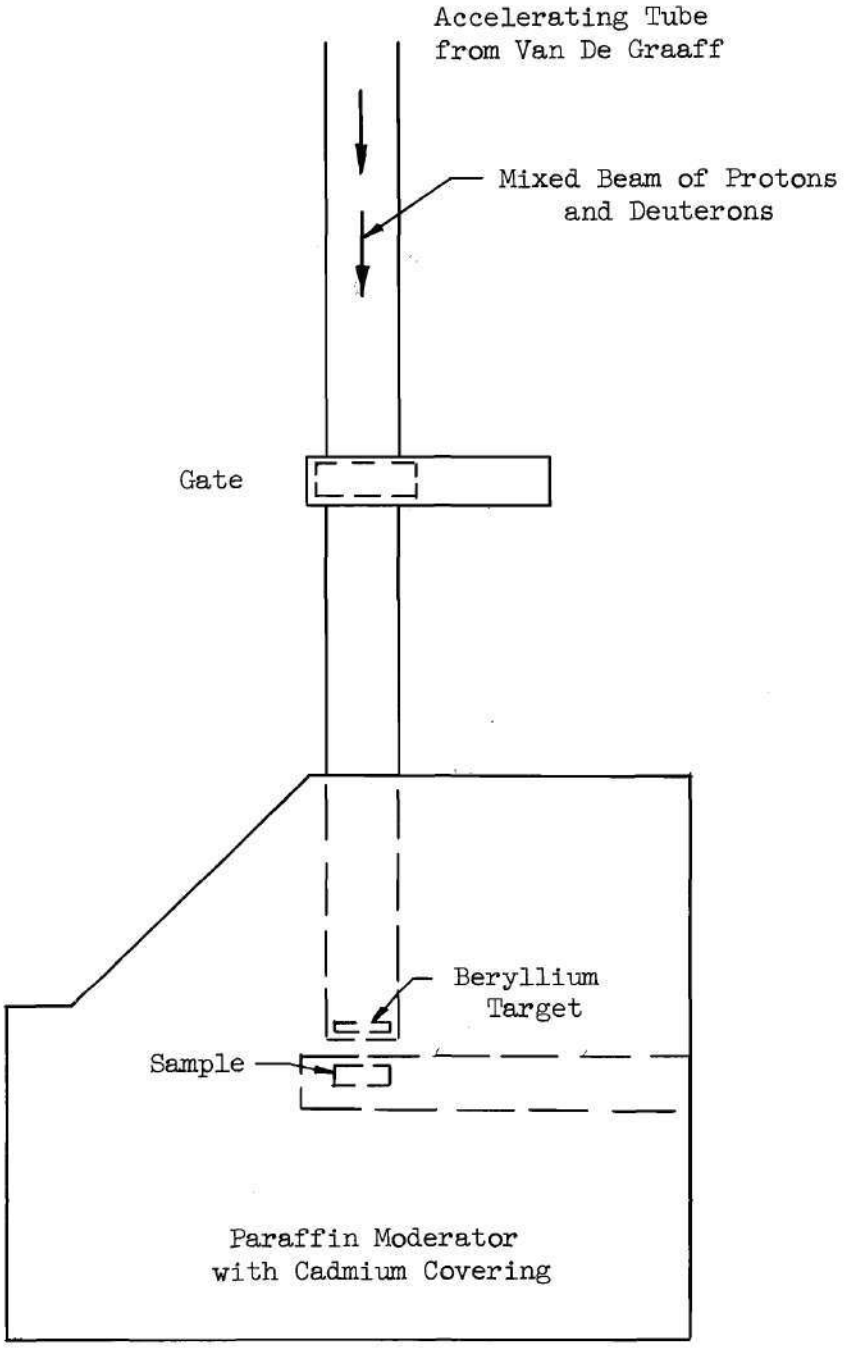

Figure 1. Thermal Neutron Irradiator

having an aluminum sleeve for positioning the mortar samples several centimeters below the target. The thermal neutron flux at the sample position will be of the order of 5×10^6 thermal neutrons per square centimeter per second.

The purpose of the paraffin is to thermalize the fast neutrons, thereby permitting their capture by the Ca^{48} atoms. The cadmium shield merely prevents the escape of thermal neutrons from the irradiator by absorption within the wall.

Radiation Detection.--To date, the use of crystals of thallium activated sodium iodide NaI(Tl) coupled to cesium-antimony phototubes is unchallenged as the most efficient method for detecting gamma rays. The following characteristics of this type of detector have resulted in a widespread application of the scintillation counter as a radiation detector and gamma ray spectrometer: high density of the inorganic crystals, which is mainly responsible for the higher stopping power and greater sensitivity to gamma rays; high light output; suitable index of refraction; response proportional to the incident radiation; and fast decay time.

The basis of a scintillation counting system is the ability of the phosphor to convert into light emissions some fraction of the energy lost by ionization during the passage of a charged particle through the material. This emitted light is picked up by the sensitive photocathode of a photomultiplier tube. The photocathode produces an electrical pulse which is similar to the light output from the crystal in both magnitude and duration. Because the electrical pulse coming from the phototube is of insufficient size to activate a scaler, additional amplification

is supplied by an external amplifier.

The greatest total efficiency in counting is obtained from having the source situated in immediate contact with the crystal and on its central axis. In this experiment, not only is this proximity very nearly attained, but also two scintillation crystals are arranged with the sample situated between them, thereby approximating four-pi geometry. With the source situated in this manner, the emissions are isotropic, and thus a large number of interactions will occur laterally in the crystals.

Figure 2 is a view of the radiation detection equipment located in the Bioengineering and Radioisotopes Laboratory at Georgia Tech. On the right, the photo shows the two scintillation crystals with lead shields mounted vertically on a small tripod. The high voltage supply is at the top of the right center instrument bank and the external linear amplifier is at the bottom. The instrument in the left center of the photo is a 100-Channel Pulse Height Analyzer (Penco) manufactured by the Pacific Electro Nuclear Co. The Penco receives the electrical pulses from the external amplifier and stores them in channels according to their individual size. The memory of the Penco is recorded on tapes by a Victor Printer shown at the right of the photo. Figure 3 is a block diagram of the pulse counting equipment.

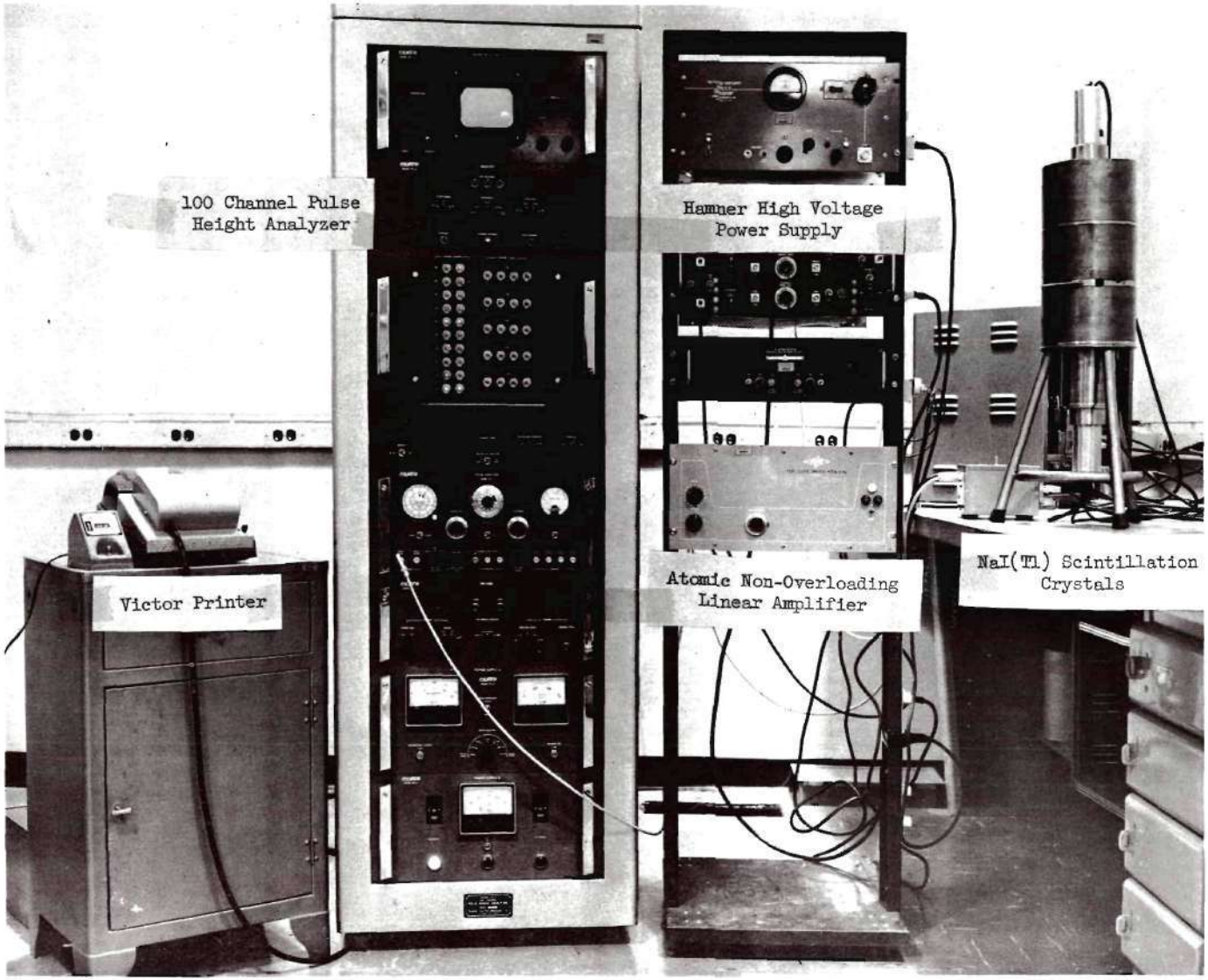

Figure 2. Instrumentation for Radiation Detection

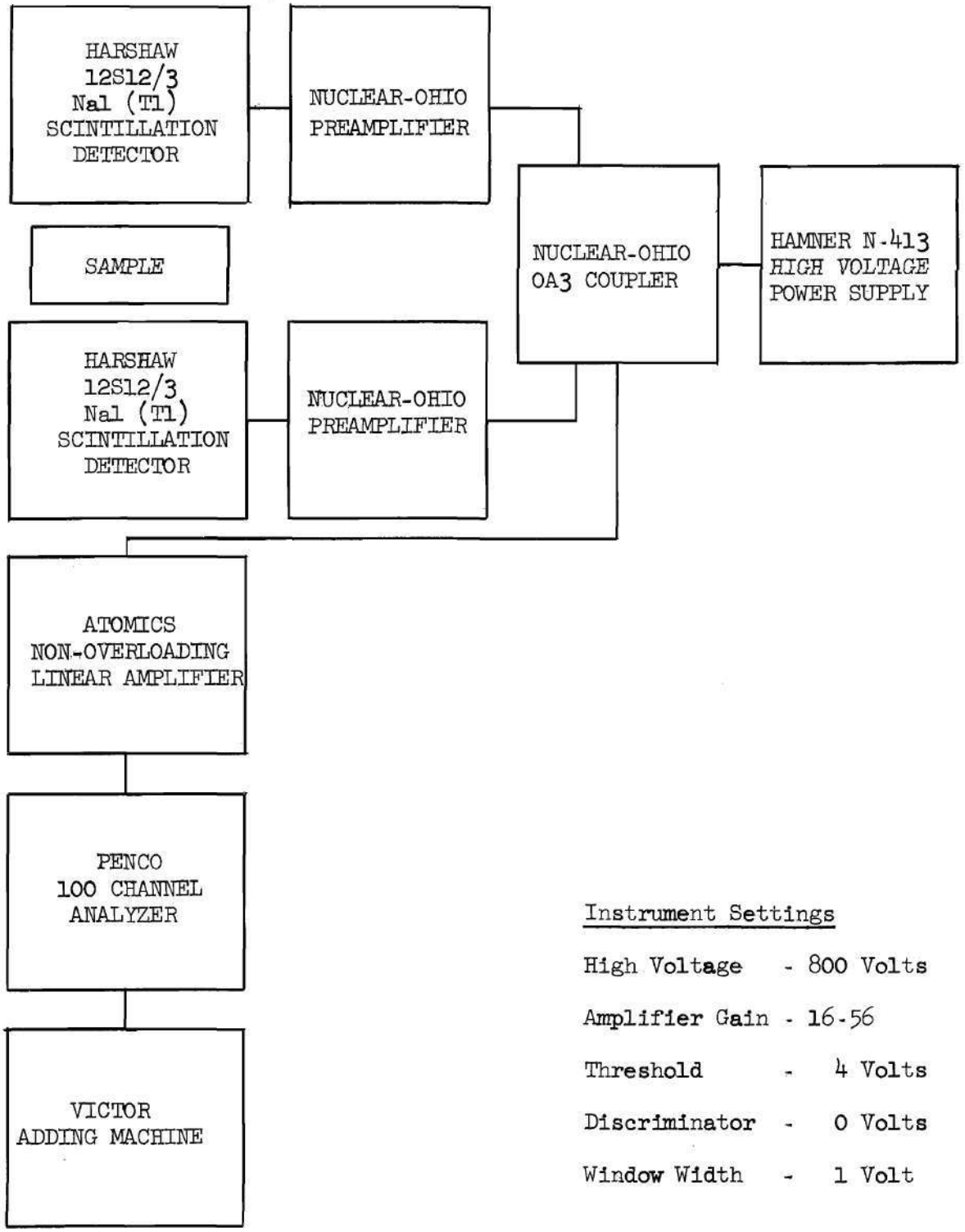

Figure 3. Schematic Diagram of Pulse Counter

CHAPTER III

DETERMINATION OF CEMENT DISPERSION

General.--In determining the dispersion of portland cement throughout a concrete mix by the use of radioisotopes, two methods are immediately available in designing the experiment.

The first method utilizes the nuclear radiations emitted from a radioactive source which has been added to the mixer. The cement is tagged with an appropriate isotope, and after predetermined periods of mixing, samples from different parts of the batch are compared for radioactivity. Two objections or obstacles arise in using this method: first, the inability to tag uniformly the large quantity of cement used in most commercial size mixers; and second, the danger of radiation exposure to plant personnel due to the dust generated during mixing of the concrete and the danger to workmen while placing radioactive concrete. These objections prevented the application of radioisotopes to the mixer.

The second method consists of activation analysis. This procedure allowed the samples to be collected without the danger of radiation exposure and to be processed in a laboratory with proper shielding and suitable monitoring devices to eliminate any health hazard. In activation, the samples to be analyzed are placed in a high flux of slow neutrons produced by the Van de Graaff for a length of time sufficient to produce a measurable amount of radioisotope of the element to be determined. The activity present is a quantitative measurement of the element. Concrete mortar samples are collected from different parts of the batch, activated,

and compared for radioactivity. Samples from the same batch showing a large variance in radioactivity would indicate insufficient mixing, while samples having a small variance in radioactivity would indicate a uniform dispersion of cement throughout the batch.

The Hapeville plant of MacDougald-Warren, Incorporated, was selected as the site for sampling because of their interest and cooperation, and because they possessed a standard type of stationary mixer used in the commercial production of ready-mixed concrete. The mixer was a 3.5-cubic-yard horizontal-tilting drum type manufactured by the T. L. Smith Corporation. Figure 4 is a picture of the plant sampled and gives the specifications for the concrete mixer.

The mixture sampled was Class "A," vibrated, air-entrained concrete, as specified by the Georgia State Highway Department. The proportions used in a 3.5-cubic-yard batch are given in Table 1.

Table 1. Proportions Used in A 3.5-Cubic-Yard Mix
Class "A," Air-Entrained Concrete

Cement	2,135 Pounds
Fine Aggregate	4,256* Pounds
Coarse Aggregate	6,282 Pounds
Water	95* Gallons
Air-Entraining Agent	14 Ounces

*Based on 4% moisture in fine aggregate and 1% moisture in coarse aggregate.

Design of Experiment.--In design of the experiment, the author consulted with Dr. Joseph Moder of the School of Industrial Engineering about the amount of data to be collected and the application of statistical concepts in the evaluation of this data. It was decided that the experiment

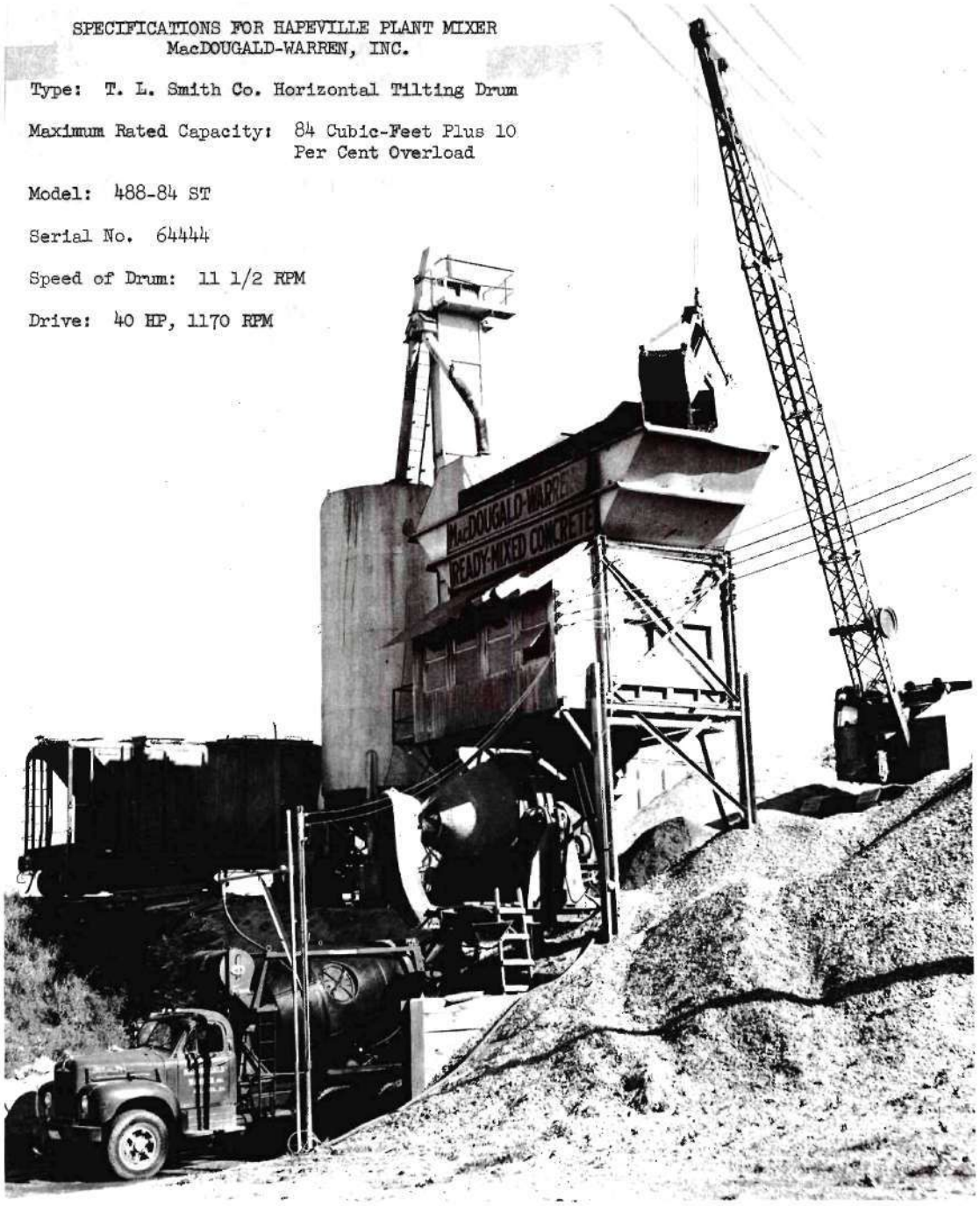

Figure 4. MacDougald-Warren Hapeville Plant

would consist of five different mix times: 30, 45, 60, 120, and 180 seconds; of collection of three samples during the discharge of each batch, which represented three different positions of the concrete in the mixer; and of evaluation of the samples for visual appearance of mixing adequacy, strength, gradation of aggregate, and cement content. The experiment was repeated twice, giving a total of forty-five samples.

The mix times throughout the experiment were randomly selected and every effort was made to eliminate systematic errors. The materials used during the test were purchased from the same suppliers; the constituents of the batch were unchanged except for minor adjustment in water; the same person did the timing throughout the tests; the collection and processing of samples were as identical as possible; and the testing procedure was not altered.

The different components of a batch were weighed and entered the mixer simultaneously. The time required for the materials to enter the mixer was between 15 and 20 seconds. In this experiment the timing of the mixing period began only after all materials had entered the mixer and continued until the concrete was to be discharged.

Because of the mixer design, it was impossible to withdraw samples directly from different locations within the mixer. Therefore, after the predetermined mixing time, the mixer was tilted, and during the 20-25 seconds necessary for discharge into a waiting truck, three samples of approximately fifty pounds each were drawn from the stream of concrete. These three samples represented the first, middle, and last third of the concrete mix being discharged. It was assumed that these samples corresponded to the concrete in the mixer near the discharge opening, in the middle third, and at the loading end.

Processing of Samples.--Immediately after being drawn, the three samples were visually graded in one of three categories: well-mixed, fair, poor (see Appendix D for criteria for classification), and then processed for future testing. Two mortar samples to be used in determining the dispersion of cement by neutron activation were collected from each of the three samples. These small samples were secured by first taking 50-100 grams of the concrete mix, removing any large aggregate by passing it through a number 4 sieve, and then filling 3/4-inch diameter by 1/4-inch high polystyrene containers with the concrete mortar. Figure 5 shows six of the mortar samples ready for the determination of cement content. Note the approximate size as compared to that of a nickel. Conventional 6-inch diameter by 12-inch high compressive strength cylinders were cast, and the remainder of each sample was used for a gradation test of the aggregate. The samples of fresh concrete used for the gradation test were washed over numbers 4, 50, and 200 sieves to remove the cement. To prevent initial setting prior to the washing described above, a retarding agent (Plastiment) was added to the samples to allow sufficient time to complete the washing process.

Testing of Samples.--To relate mixing time to uniformity of a concrete mix, strength and gradation of aggregate were recorded, as well as the dispersion of cement. Tests were first run for determining the uniformity of the aggregate in each sample. After being washed, the aggregate was dried at 235° F. for 24 hours and its fineness modulus determined by running a sieve analysis test.[6] Twenty-eight day compressive strength

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[6]Fineness modulus is a numerical coefficient used to describe the sieve analysis of an aggregate. The percentage of material coarser than each sieve size is calculated, and the sum of these percentages divided by 100 is the fineness modulus. The larger the aggregate, the higher is its fineness modulus.

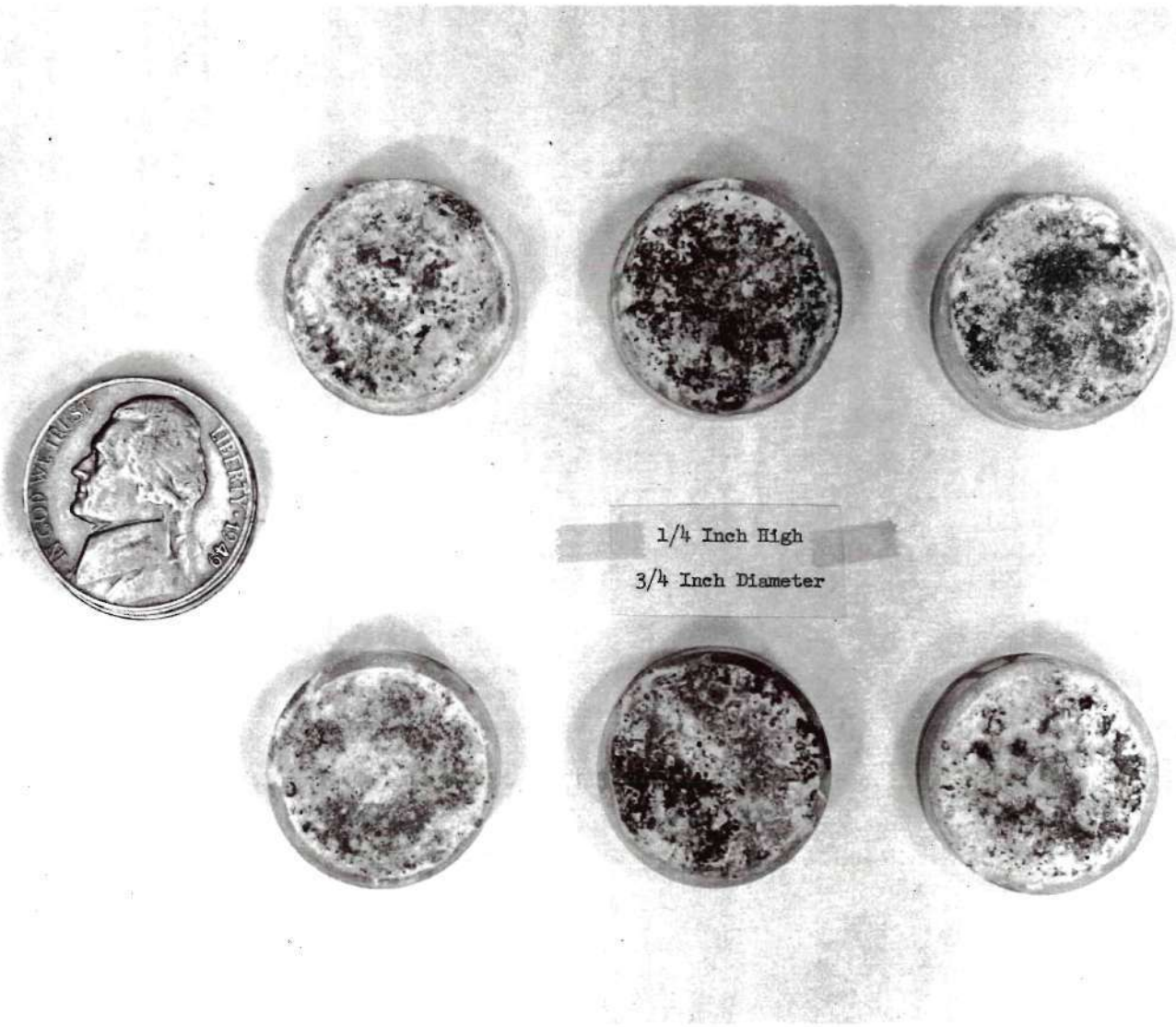

Figure 5. Concrete Mortar Samples

of the concrete was determined from the 6 by 12-inch cylinders under testing conditions prescribed by the American Society of Testing Materials (C85-54).

The most difficult evaluation to make was the determination of the cement content of the cast mortar samples. As stated previously, irradiation can make the sample radioactive, and a knowledge of the particle emissions from the sample then allows interpretation of the sample's composition.

The problem consisted of finding an element within the portland cement that was not present in the other constituents of the concrete batch. Table 2 lists the chemical properties of the cement and aggregate used.

By weight, calcium oxide comprises about 65 per cent of portland cement. For the material used in this experiment, calcium is only present in a very small percentage in the coarse aggregate, and is not found at all in the fine aggregate. Because only 1 per cent of the coarse aggregate passes a number 4 sieve, only a minute fraction of the calcium present in a mortar sample would be contributed by the coarse aggregate. For this reason, radiation of calcium was selected as the cement content index.

An investigation of calcium was made to determine whether an isotope existed which, when subjected to neutron activation, would become traceable. It was also necessary to examine isotopes of the other chemical elements constituting concrete to insure that their energy of radiation and disintegration did not interfere with calcium measurements. The results of this investigation are shown in Table 3.

Table 2. Chemical Analysis of Portland Cement and Fine and Coarse Aggregate

Chemical Compound	Per Cent by Weight
Portland Cement	
Universal Atlas, Birmingham, Alabama	
CaO	65.66
SiO_2	22.24
Al_2O_3	5.96
Fe_2O_3	2.16
SO_3	1.88
MgO	0.93
Ins. Res.	0.40
K_2O	0.15
Na_2O	0.03
Fine Aggregate	
Alluvial Deposit Known as the Tuscaloosa Formation	
Taylor Sand Co., Junction City, Georgia	
SiO_2	98.00
Al_2O_3	1.20
H_2O	0.56
Org. Matter	0.18
Fe_2O_3	0.06
Coarse Aggregate	
Biotite Granite Gneiss	
Tyrone Rock Products Co., Quarry No. 2, Mt. View, Georgia	
SiO_2	74.70
Al_2O_3	13.92
Fe_2O_3	3.84
CaO_3	3.76
Na_2O	2.80
K_2O	0.76
MgO	0.20

Table 3. Isotopes Inherent in Elements of Concrete

Target Isotope	Abundance (%)	Radio-Nuclei	Type of Decay	Half-Life	Energy of Radiation and Disintegration (Mev)
$_1H^2$	0.015	$_1H^3$	β^-	12.26 Yrs.	0.18
$_8O^{18}$	0.204	$_8O^{19}$	β^-	29.4 Sec.	4.5, (30%)
					2.9, (70%)
			γ		1.6, (70%)
$_{11}Na^{23}$	100.	$_{11}Na^{24}$	β^-	14.97 Hrs.	4.122, (100%)
					4.17, (.003%)
			γ		1.380, 2.758
$_{12}Mg^{26}$	11.29	$_{12}Mg^{27}$	β^-	9.45 Min.	1.75, (58%)
					1.59, (42%)
			γ		0.834, 1.015
$_{13}Al^{27}$	100.	$_{13}Al^{28}$	β^-	2.27 Min.	2.87, (100%)
			γ		1.78
$_{14}Si^{30}$	3.05	$_{14}Si^{31}$	β^-	2.62 Hrs.	1.49
			γ		1.264, (.07%)
$_{16}S^{34}$	4.215	$_{16}S^{35}$	β^-	87 Days	0.167, (100%)
$_{16}S^{36}$	0.017	$_{16}S^{37}$	β^-	5.04 Min.	1.6, (90%)
					4.3, (10%)
			γ		3.09
$_{19}K^{39}$	93.08	$_{19}K^{40}$	β^-	1.25 x 10^9 Yrs.	1.33, (89%)
					1.46, (11%)
			Electron Capture		
$_{19}K^{41}$	6.91	$_{19}K^{42}$	β^-	12.52 Hrs.	2.04, (25%)
					3.58, (75%)
			γ		1.51, (20%)
$_{20}Ca^{44}$	2.06	$_{20}Ca^{45}$	β^-	164 Days	0.254
$_{20}Ca^{46}$	0.0033	$_{20}Ca^{47}$	β^-	4.7 Days	0.70, (76%)
					1.94, (24%)
			γ		0.50, (5%)
					0.81, (5%)
					1.29, (71%)
$_{20}Ca^{48}$	0.185	$_{20}Ca^{49}$	β^-	8.9 Min.	1.0, 2.12
			γ		3.07, (89%)
					4.04, (10%)
					4.7, (0.8%)
$_{26}Fe^{54}$	5.84	$_{26}Fe^{55}$	Electron Capture	2.60 Yrs.	
$_{26}Fe^{58}$	0.31	$_{26}Fe^{59}$	β^-	45.1 Days	0.271, (46%)
					0.462, (54%)
			γ		1.560, (.3%)
					1.099, (57%)
					1.289, (43%)

Although not in great abundance, $_{20}Ca^{48}$ was selected as the target nuclei which, after activation in the Van de Graaff, becomes the radioactive isotope $_{20}Ca^{49}$. This isotope was selected because of its relatively short half-life and traceable energy emissions during decay.

When bombarded by thermal neutrons, $_{20}Ca^{48}$ undergoes the following transformation:

$$_{20}Ca^{48} + {}_{0}n^{1} \rightarrow {}_{20}Ca^{49}$$

$_{20}Ca^{49}$ has been found to decay with a half-life of 8.9 ± 0.2 minutes. The decay scheme, as determined by Martin, Cork, and Burson,[7] is shown in Figure 6.

The gamma ray spectrum of an activated cement mortar sample was studied to insure that the 3.07-Mev (million-electron-volts) gamma ray emitted by Ca^{49} could be detected using the two 3-inch diameter NaI (Tl) crystals and the Penco 100-Channel Pulse Height Analyzer. The spectrum of an activated cement mortar sample as determined by the scintillation counting system is shown in Figure 7.

First, a calibration of the 100 channel analyzer was necessary to determine in which channel the 3.07-Mev sum-peak would fall. Sources with known energy emissions were counted and plotted against channel number to give a calibration curve. The channel numbers corresponding to the different energy peaks are given in Table 4. From the calibration curve shown in Figure 8, it was possible to select the approximate channel in which a 3.07-Mev energy pulse would fall.

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[7] D. W. Martin, J. M. Cork, and S. B. Burson, "Decay of Ca^{49} and Sc^{49}," The Physical Review, Vol. 102, No. 2, April 15, 1956, pp. 457-458.

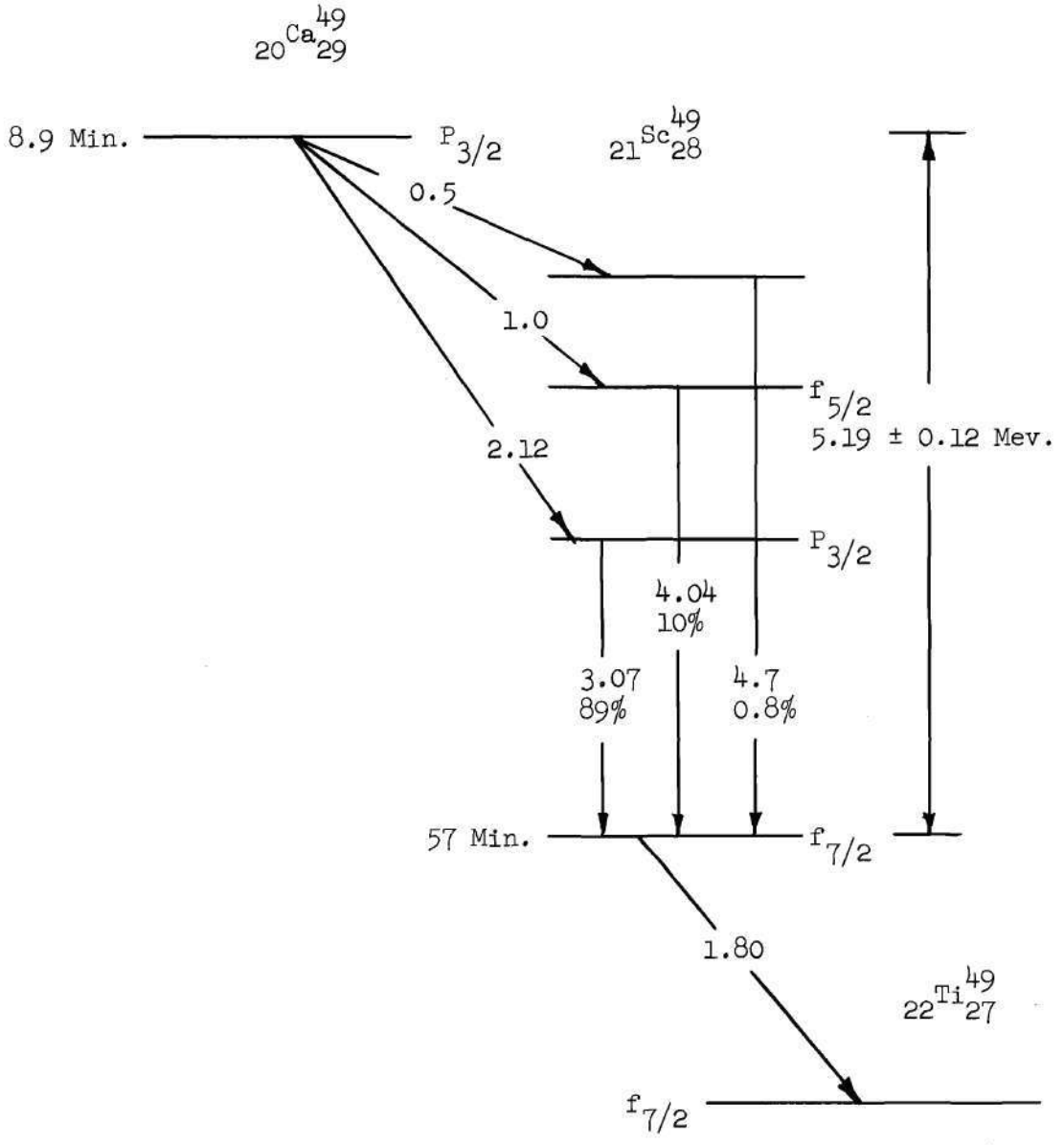

Figure 6. Decay Scheme of Calcium 49 and Scandium 49

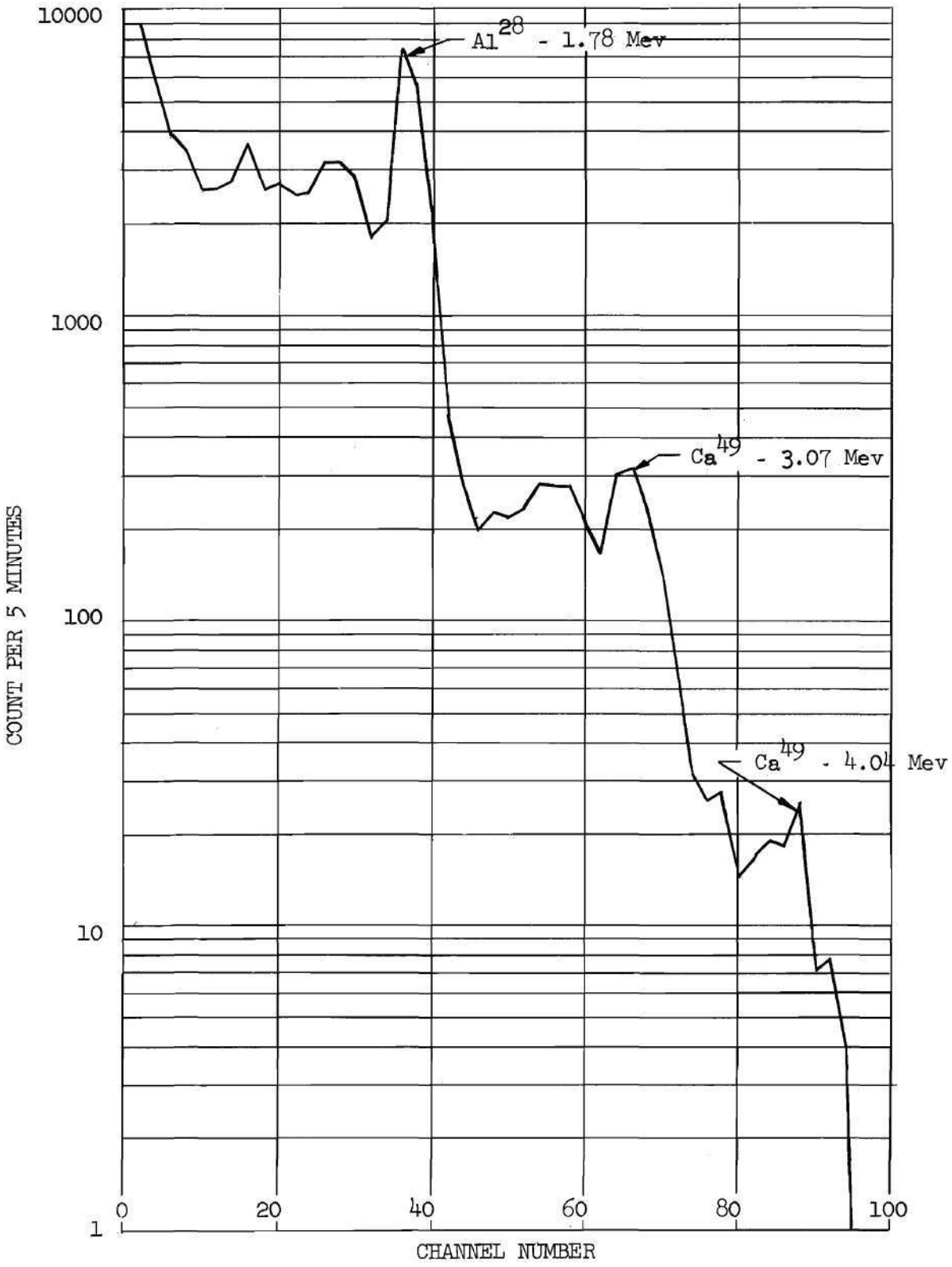

Figure 7. Gamma Ray Spectrum of Activated Mortar Sample

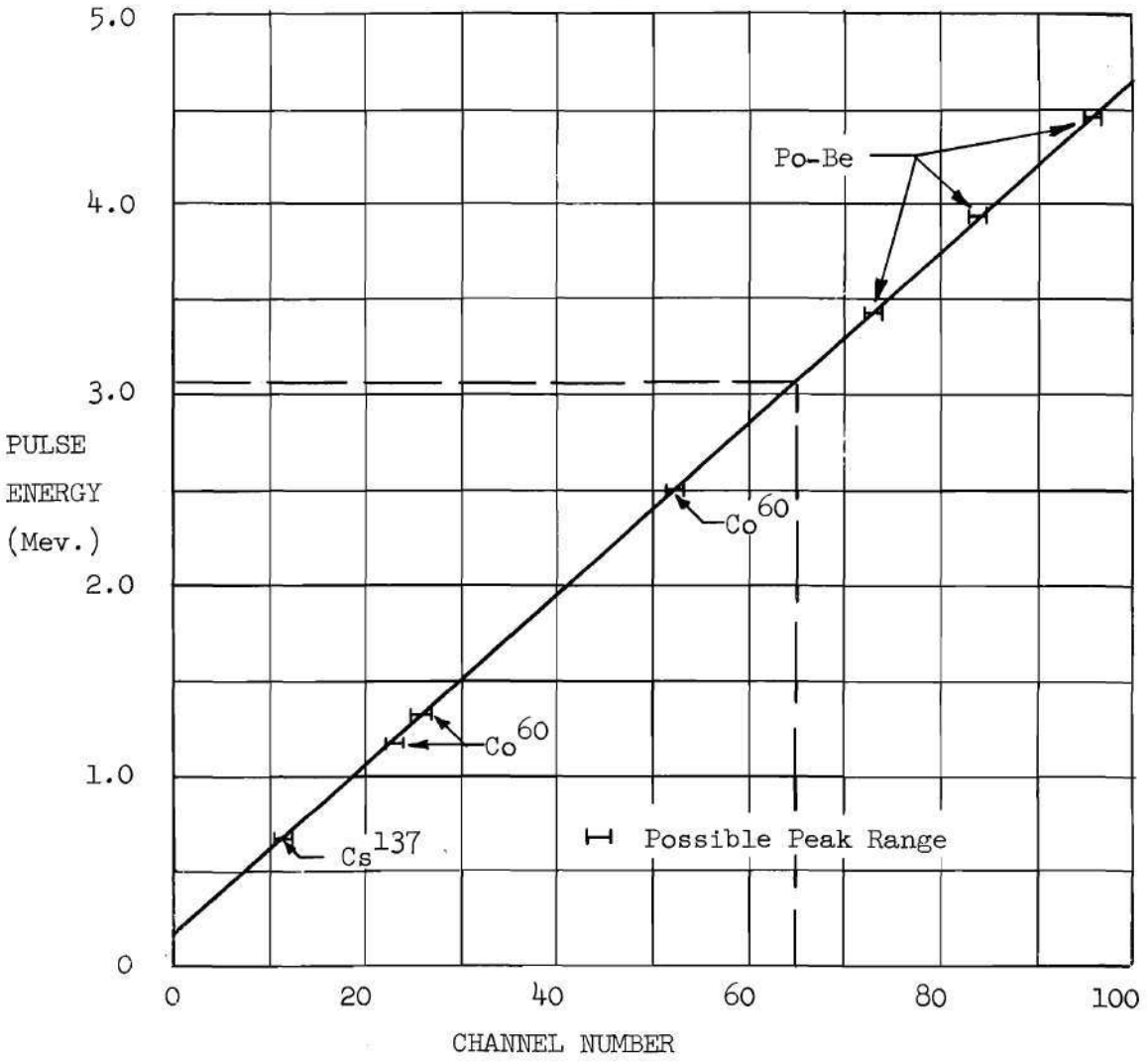

Figure 8. Calibration Curve for 100 Channel Pulse Height Analyzer

Table 4. Gamma Sources for Penco Calibration

Isotope	Energy Peaks	Channel No.
Cs^{137}	0.667	11 1/2
Co^{60}	1.17	23
	1.33	26
	2.50	52 1/2
Po-Be	3.43	73
	3.94	84
	4.45	96

A decay study was then run on the 3.07-Mev energy peak of an activated mortar sample as a check of its half-life. After activation, the sample was transferred to the scintillation counter and three 5-minute counts were recorded. The peak was found to cover channels 62-74. Table 5 gives the total counts recorded under the 3.07-Mev peak at the end of each counting period.

Table 5. Decay Study of 3.07-Million-Electron-Volt Energy Peak

Time after End of Irradiation (Minutes)	Counts, Channels 62-74
1-6	2,409
9-14	1,242
17-22	648

Figure 9 is a partial plot of the spectrum at the end of each 5-minute counting period; this plot graphically shows the decay of the

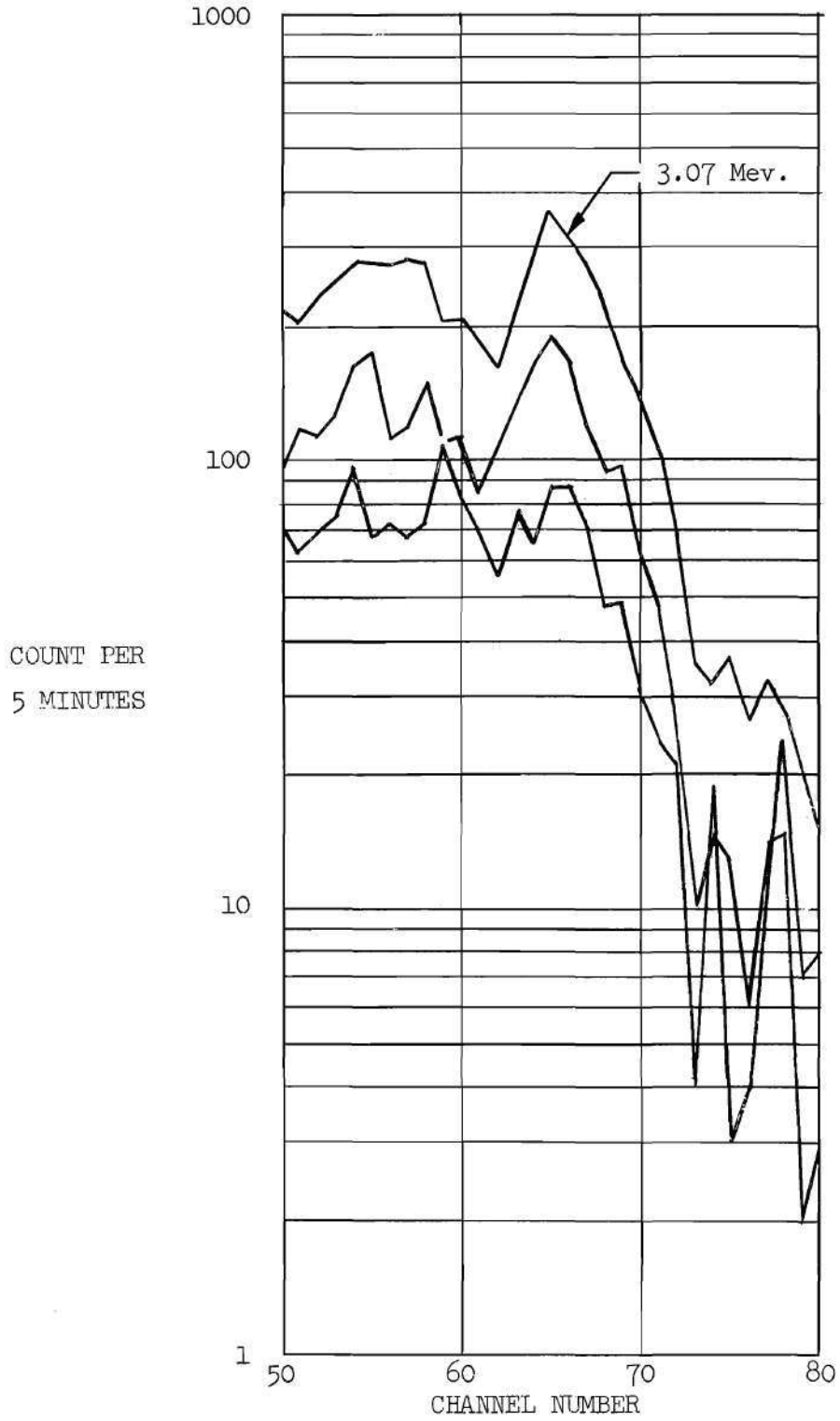

Figure 9. Decay of 3.07-Million-Electron-Volt Energy Peak

3.07-Mev energy peak. Figure 10 shows the total counts recorded in channels 62-74 for the three 5-minute counts spaced 8 minutes apart. From Figure 10, the half-life of the peak was determined by reading the time on the abscissa corresponding to a 50 per cent reduction in activity on the ordinate scale.

A plot of the data determined the half-life of the peak to be 8.5 minutes. The difference of 0.4 minutes between theoretical and observed decay time for $_{20}Ca^{49}$ was due to the presence of $_{16}S^{37}$.

When irradiated, $_{16}S^{36}$ nuclei enter the excited state of $_{16}S^{37}$ and decay, emitting 3.09-Mev gamma rays with a half-life of 5.04 minutes. Sulfur ionization therefore contributes a few of the counts recorded in the 3.07-Mev peak of the spectrum, but this should in no way reduce the accuracy in cement content determination, since sulfur is only present in the cement.

With the half-life of $_{20}Ca^{49}$ known to be 8.9 minutes, it was decided that an irradiation period of 10 minutes for the collected mortar samples would give a sufficient number of counts to determine adequately the cement content. During this period the increase in activity is nearly linear with the time of irradiation.

The intensity of the 25 micro ampere beam varies during this 10-minute period, among different samples, and from day to day. Therefore, it was necessary to monitor the varying neutron flux with small pieces of indium foil which were irradiated along with each sample. The counts obtained from the indium foils were first normalized to correct for the varying foil weights, and then further normalized to correct for the variation in neutron flux. The count of each mortar sample could

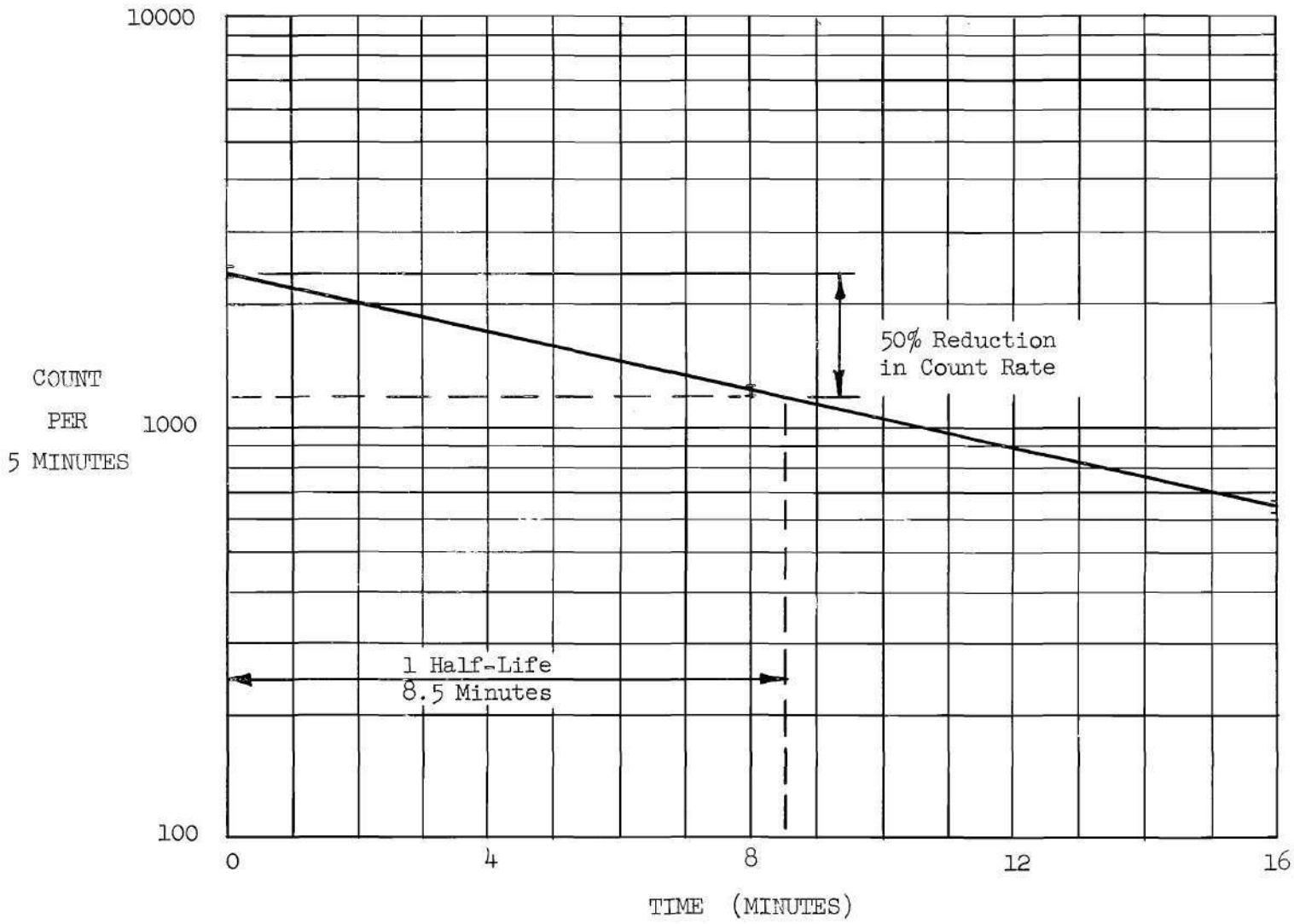

Figure 10. Half-Life Determination of 3.07-Million-Electron-Volt Peak

then be normalized to the value it would have recorded had the neutron flux been constant during the testing period. An example of the calculations necessary to correct each sample's count is given in Appendix C.

After a 10-minute exposure to the thermal neutron flux, the samples were removed from the irradiator and taken to the counting room. One minute was allowed for the transfer of a sample and an additional 30 seconds for transferring the monitoring indium foil, with each then being counted for 5 minutes.

The outputs from the two photomultiplier tubes were added electronically, giving a single composite spectrum which may be seen on the Penco pulse height analyzer scope. The spectrum was then printed on tape to give a permanent record of each sample's activity. The observed 5-minute count of the indium foil was recorded by a Geiger-Mueller Counter.

To determine the cement content of mortar samples collected at the MacDougald-Warren plant, a standard cement-content-versus-count chart or graph was developed. Laboratory samples were made with known quantities of cement were activated, counted, and plotted. Because of the random decay of radioactive isotopes, three observations were made of each standard sample and the best line through the points was determined by the method of least squares (see Figure 11). It was from this line that point estimates of the cement content of cast mortar samples were determined.

CHAPTER IV

RESULTS

Analysis of Variance.--The analysis of variance is probably the most powerful procedure in the field of experimental statistics. It allows the data collected to be rigorously analyzed and our conclusions to be accompanied by probability statements as to the correctness of our inferences. To carry out the analysis, it is necessary to formulate a mathematical model in terms of the unknown parameters and the associated random variables. The quantitative physical characteristics (dependent variables) of interest in this study are the following:

(1) Aggregate fineness modulus

(2) Compressive strength

(3) Cement content of mortar

(4) Visual evaluation of mixing

Aggregate fineness modulus and cement content may appear to be independent variables since they are set by the particular mix sampled, but in this investigation where the constituents enter the mixer in segregated slugs and this thesis is concerned with the dispersion of the constituents, aggregate fineness modulus and cement content are dependent variables.

The independent variables of interest are as follows:

(1) Mixing time (T) 30, 45, 60, 120, and 180 seconds

(2) Position of concrete in discharge stream or in mixer

(3) Replication (experiment is run three times)

The following analysis was made on the strength and fineness modulus characteristics, and on the average of the two cement content determinations for each sample:

Table 6. Primary Variables

Factor	Abbreviation	Subscript	No. Levels	Model
Mix Time	T	i	5	Fixed
Replication	R	j	3	Fixed
Discharge Position	P	k	3	Fixed

The mathematical model can be written as

$$y_{ijk} = \mu + T_i + R_j + RT_{ij} + P_k + PR_{jk} + PT_{ik} + PRT_{ijk}$$

In effect, this formula states that for an individual concrete sample, the strength, fineness modulus, or cement content (determination) for the kth position in the jth replication, mixed for ith seconds, will be an expected value μ, plus the sum of any main effects and interaction effects due to the three independent variables.

Table 7 gives the design used for the analysis of variance, which is known as a Split-Plot experiment. RT is used as an estimate of $\sigma_{\epsilon 2}^2$, the main plot error, and PRT is taken as an estimate of $\sigma_{\epsilon 1}^2$, the split-plot error.

Fineness Modulus.--Table 8 gives the aggregate fineness modulus of the forty-five samples collected during the experiment. Results of the analysis of variance are shown in Table 9.

Table 7. Source of Variation, Degrees of Freedom, Sum of Squares, and Expected Mean Squares for Split-Plot Experiment

Source of Variation	Degrees of Freedom	Sum of Squares	Expected Mean Square
R	2	$\Sigma T_{.j.}^2/15 - T_{...}^2/45$	$\sigma_{\epsilon_2}^2 + 15\theta_R^2$
T	4	$\Sigma T_{i..}^2/9 - T_{...}^2/45$	$\sigma_{\epsilon_2}^2 + 9\theta_T^2$
RT	8	$\Sigma\Sigma T_{ij.}^2/3 - \Sigma T_{i..}^2/9 - \Sigma T_{.j.}^2/15 + T_{...}^2/45$	$\sigma_{\epsilon_2}^2$
P	2	$\Sigma T_{..k}^2/15 - T_{...}^2/45$	$\sigma_{\epsilon_1}^2 + 15\theta_P^2$
PR	4	$\Sigma\Sigma T_{.jk}^2/5 - \Sigma T_{.j.}^2/15 - \Sigma T_{..k}^2/15 + T_{...}^2/45$	$\sigma_{\epsilon_1}^2 + 5\theta_{PR}^2$
PT	8	$\Sigma\Sigma T_{i.k}^2/3 - \Sigma T_{i..}^2/9 - \Sigma T_{..k}^2/15 + T_{...}^2/45$	$\sigma_{\epsilon_1}^2 + 3\theta_{PT}^2$
PRT	16	$\Sigma\Sigma\Sigma y_{ijk}^2 - \Sigma\Sigma T_{ij.}^2/3 - \Sigma\Sigma T_{i.k}^2/3 - \Sigma\Sigma T_{.jk}^2/5 - T_{...}^2/45 + \Sigma T_{i..}^2/9 + \Sigma T_{.j.}^2/15 + \Sigma T_{..k}^2/15$	$\sigma_{\epsilon_1}^2$
Total	44	$\Sigma\Sigma\Sigma y_{ijk}^2 - T_{...}^2/45$	

Table 8. Aggregate Fineness Modulus

Replication	Position in Mixer or Discharge Stream	Mixing Time (Seconds)				
		30	45	60	120	180
1	1	4.61	4.78	4.72	5.03	5.09
	2	5.09	5.05	5.09	5.01	4.96
	3	5.22	4.98	5.09	5.17	5.09
2	1	4.76	4.49	4.76	4.99	4.31
	2	5.01	4.85	5.05	5.07	5.17
	3	5.07	4.96	4.78	4.96	5.30
3	1	4.73	4.69	4.68	4.32	4.96
	2	5.00	4.91	4.94	5.15	5.08
	3	5.16	4.81	5.00	5.27	4.97

Table 9. Analysis of Variance for Fineness Modulus

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Tests		
				F	$F_{0.05}$	$F_{0.01}$
Replication	0.0853	2	0.0427	7.91*	4.46	8.65
Mix Time	0.1689	4	0.0422	7.81	3.84	7.01
RT	0.0428	8	0.0054			
Position	0.9913	2	0.4957	9.80**	3.63	6.23
PR	0.0455	4	0.0114	0.23	3.01	4.77
PT	0.0509	8	0.0064	0.13	2.59	3.89
PRT	0.8090	16	0.0506			
Total	2.1939	44				

*Significant at the 5% level
**Significant at the 1% and 5% levels

In checking whether the variances were equal among the five mixing times, i.e., whether $\sigma_{30}^2 = \sigma_{45}^2 = \cdots \sigma_{180}^2$, Bartlett's test gave no evidence of any difference among σ^2's at either the 1 or the 5 per cent levels.

From Table 9 it may be noted that none of the interaction terms are significant at the 1 or 5 per cent level, while all main effects had F-ratios in excess of $F_{0.05}$. Thus it may be concluded that the mix times, replications, and positions used in this experiment do contribute significantly to the variation in the fineness modulus.

At the 1 per cent level, both the mix times and the positions were significant, but the three replications were not found to affect the variation in fineness modulus.

In rejecting the hypothesis that the position of the concrete in the discharge stream does not affect the fineness modulus, it is possible to determine which positions differ. By the application of Tukey's procedure of contrasts,[8] one can conclude that the fineness modulus in Position 1 differs significantly from the fineness modulus in Positions 2 and 3, and that there is no significant difference between the fineness modulus in Positions 2 and 3.

Compressive Strength.--Compressive strength is universally used as the index of concrete quality, but, used alone, it may be misleading. Samples drawn from two different batches of concrete may exhibit similar strength even though their degree of mixing is quite different. A sample

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[8] Albert H. Bowker and Gerald J. Lieberman, "Analysis of Variance," Engineering Statistics, 1959, p. 295.

with inadequate moisture content may be unacceptable from the standpoint of workability; yet, may give high strength after being cast in a cylinder mold.

Table 10 shows the compressive strengths obtained in breaking tests on the 6 by 12-inch concrete cylinders. The analysis of variance computed for compressive strength of concrete is given in Table 11. Bartlett's test indicated that the variances among mix times were equal at both the 1 and the 5 per cent levels.

The value of F obtained in testing the hypothesis that the different replications do not affect the strength was 5.28. Even though this value exceeds $F_{0.05} = 4.46$, the author believes this was a chance error, since no assignable reason for the error variance is evident.

In the past, numerous articles have been published correlating the strength of concrete with mixing time (1, 2, 8, 19). It is the present consensus that 1 minute is the minimum length of time for suitable mixing of concrete, and that 2 minutes is highly desirable. The value of F computed in the analysis of variance for testing the mixing time effect indicated there was not a significant difference in strength for different mixing times.

Immediately evident is the highly significant value of F = 12.23 for testing the position hypothesis. It can be observed that from the data of this experiment the strength of concrete varied significantly among each of the three positions in the discharge stream. Observing the F values computed in Table 11, there is no reason to doubt the hypothesis that there is no significant difference in strength because of interaction effect among the independent variables.

Table 10. 28-Day Compressive Strength in Pounds Per Square Inch of Class "A" Concrete

Replication	Position in Mixer or Discharge Stream	Mixing Time (Seconds)				
		30	45	60	120	180
1	1	5850	5210	6230	5180	4730
	2	4210	4980	5210	4720	3980
	3	2650	4360	3880	3890	4330
2	1	5420	5050	5040	3830	3570
	2	5110	5370	4880	3300	2810
	3	3230	4220	3760	3600	3000
3	1	5390	6170	3220	3750	3270
	2	4380	4650	3040	3290	3390
	3	2190	3250	3220	2560	3000

Table 11. Analysis of Variance for Compressive Strength

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Tests		
				F	$F_{0.05}$	$F_{0.01}$
Replication	71,448	2	35,724	5.28*	4.46	8.65
Mix Time	83,942	4	20,986	3.10	3.84	7.01
RT	54,106	8	6,763			
Position	145,230	2	72,615	12.23**	3.63	6.23
PR	6,061	4	1,515	0.25	3.01	4.77
PT	57,222	8	7,153	1.20	2.59	3.89
PRT	94,969	16	5,936			
Total	512,978	44				

*Significant at the 5% level
**Significant at the 1% and 5% levels

Cement Content.--It is known that the fluctuations associated with the decay of radioactive material are properly described by the Poisson-distribution law, but because it is relatively easy to handle mathematically, the normal-distribution law is frequently used as an approximation. According to the normal-distribution law, positive and negative deviations of a given magnitude are equally likely, and the occurrence of large deviations is less probable than that of small ones.

In the determination of cement content of a mortar sample, it was necessary to predict the cement content from a count rate subject to statistical variation. Therefore, it is necessary to indicate the reliability of any results reported.

Since there is an underlying physical relationship between observed count and cement content, it is appropriate to make point estimates of the cement content associated with a particular count. But since the observed count contains an error component, a confidence interval estimate is also needed to enable probability statements to be made about the true cement content of the samples.

For this experiment, a 95 per cent confidence interval was chosen in determining cement content. A sample having a corrected count of 600 between channels 62-74 would have a point estimate equal to 0.508 gram of cement and a 95 per cent confidence interval equal to $\pm$0.068 gram. The point estimate for a sample recording 1,800 counts per 5 minutes would equal 1.592 grams of cement and a 95 per cent confidence interval of $\pm$0.066 gram or a range in cement content from 1.536 to 1.658 grams.

Figure 11 shows the cement-content-versus-count-line and gives its equation and correlation coefficient r. The value of r = 0.997 indicates a nearly perfect degree of association between the two related variables.

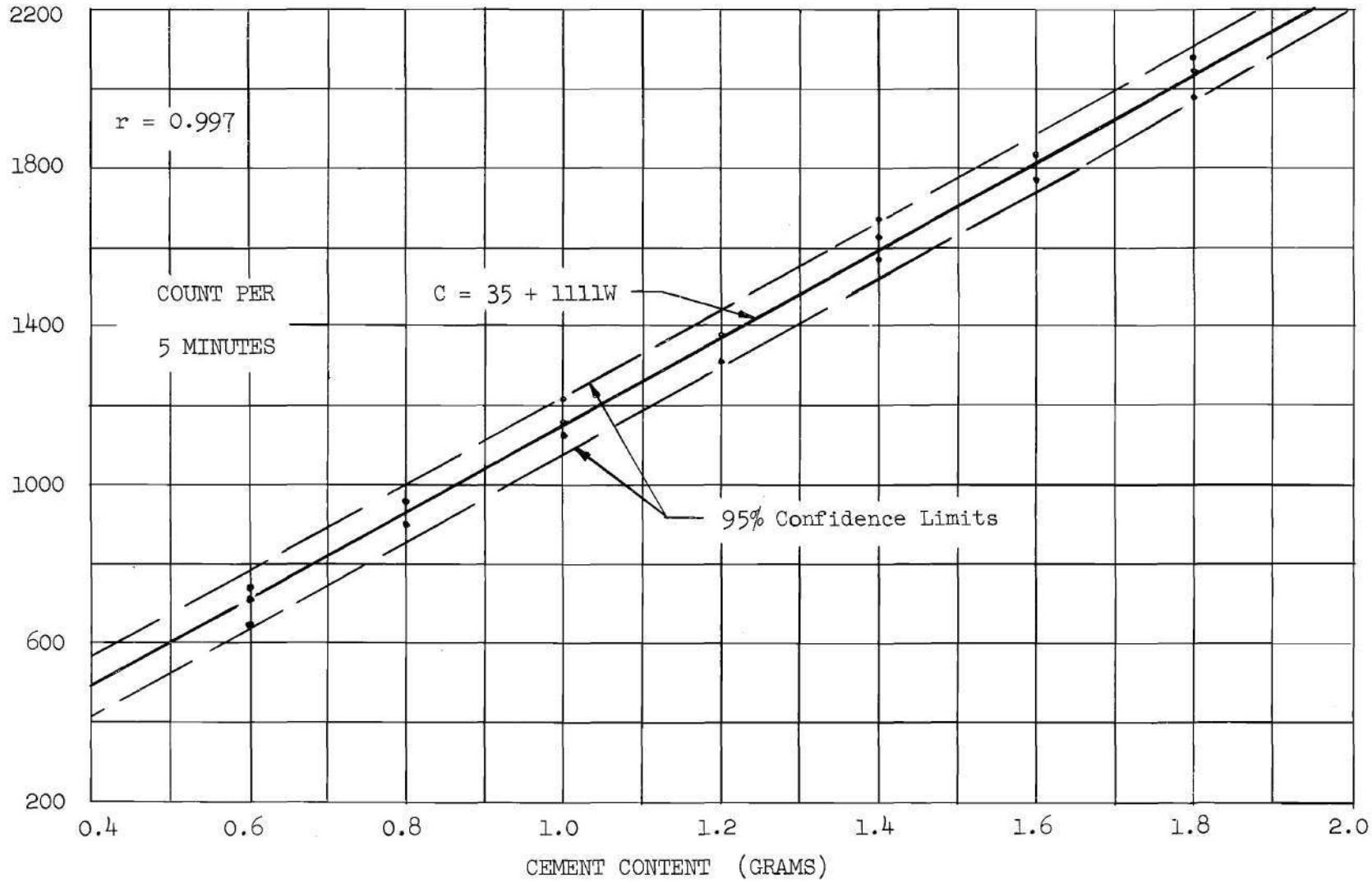

Figure 11. Cement-Content-Versus-Count-Line

The 95 per cent confidence limits are also shown in Figure 11.

Table 12 gives the point estimates of the cement content of the ninety mortar samples tested. The analysis of variance for cement content determination is given in Table 13. The hypothesis that $\sigma_{30}^2 = \sigma_{45}^2 = \cdots \sigma_{180}^2$ was accepted when Bartlett's test gave no reason to doubt the prior assumption.

For testing the hypothesis that the position of the concrete in the discharge stream has no effect on the cement content of a sample, the value of F was 14.69. Since this value exceeds both $F_{0.05} = 3.63$ and $F_{0.01} = 6.23$, one can conclude that there is a highly significant variation in cement content of mortar among samples located in different positions in the discharge stream or its corresponding position in the mixer.

Using Tukey's procedure of contrasts, it may be shown that the cement content of Position 1 samples differ significantly from those in Positions 2 and 3, and that there is no significant difference between the cement contents of Positions 2 and 3.

Referring to the other values of F in Table 13, the main effects and interaction effects among the variables were insufficient to cause the cement content to differ significantly.

Visual Inspection.--An objective type of evaluation was attempted to determine whether the author could visually ascertain the degree of mixing by inspecting the samples of concrete discharged from the mixer. The forty-five samples used in the previously described tests were classified in one of three categories: well-mixed, fair, and poor.

Compressive strength, fineness modulus, and cement content of

Table 12. Cement Content of Mortar Samples in Grams Cement Per Gram Mortar

Replication	Position in Mixer or Discharge Stream	Mixing Time (Seconds)				
		30	45	60	120	180
1	1	.22	.20	.32	.27	.32
		.22	.22	.36	.33	.30
	2	.12	.28	.20	.26	.23
		.22	.21	.23	.29	.29
	3	.16	.20	.18	.25	.20
		.18	.21	.26	.18	.28
2	1	.35	.33	.25	.21	.21
		.33	.33	.23	.24	.28
	2	.27	.21	.25	.24	.23
		.32	.30	.29	.25	.25
	3	.24	.19	.19	.18	.14
		.32	.31	.21	.23	.21
3	1	.35	.43	.29	.32	.27
		.34	.36	.32	.32	.13
	2	.18	.16	.20	.23	.15
		.25	.19	.24	.20	.20
	3	.20	.21	.22	.23	.22
		.20	.24	.28	.18	.23

Table 13. Analysis of Variance Table for Cement Content

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Tests		
				F	$F_{0.05}$	$F_{0.01}$
Replication	0.0013	2	.000650	0.14	4.46	8.65
Mix Time	0.0033	4	.000825	0.18	3.84	7.01
RT	0.0367	8	.004587			
Position	0.0435	2	.02175	14.69**	3.63	6.23
PR	0.0128	4	.0032	2.16	3.01	4.77
PT	0.0049	8	.000612	0.41	2.59	3.89
PRT	0.0237	16	.001481			
Total	0.1262	44				

**Significant at the 1% and 5% levels

samples were used to correlate visual classification to degree of mixing. Table 14 ranks the forty-five samples according to their compressive strength and gives each sample's visual estimate of mixing uniformity. Only three of the samples were not considered to be well-mixed, and these were given a fair rating.

Since the three samples classified as being mixed to a fair degree were from the same batch, it was decided to rank the 15 batches according to the range of compressive strength, fineness modulus, and cement content found within each batch. Tables 15, 16, and 17 show the location of the batch containing the three "fair-mixed" samples in the three rankings above.

Although an analysis of variance can not be performed on this data, it may be concluded from an observation of the data that the author was unable to determine visually the degree of mixing uniformity. In Table 14, one of the samples having a fair rating fell within each third of the strength rank, and in Tables 15, 16, and 17 there were always batches classified as well-mixed that had greater ranges of compressive strength, fineness modulus, and cement content among the three samples than that of the one batch in question.

Table 14. Compressive Strength and Visual Classification of Concrete Samples

Sample Number			Compressive Strength (Lbs./Sq. In.)	Degree of Mixing A, B, C*
Mix Time (Seconds)	Replication	Position		
60	1	1	6230	A
45	3	1	6170	A
30	1	1	5850	A
30	2	1	5420	A
30	3	1	5390	B
45	2	2	5370	A
45	1	1	5210	A
60	1	2	5210	A
120	1	1	5180	A
30	2	2	5110	A
45	2	1	5050	A
60	2	1	5040	A
45	1	2	4980	A
60	2	2	4880	A
180	1	1	4730	A
120	1	2	4720	A
45	3	2	4650	A
30	3	2	4380	B
45	1	3	4360	A
180	1	3	4330	A
45	2	3	4220	A
30	1	2	4210	A
180	1	2	3980	A
120	1	3	3890	A
60	1	3	3880	A
120	2	1	3830	A
60	2	3	3760	A
120	3	1	3750	A
120	2	3	3600	A
180	2	1	3570	A
180	3	2	3390	A
120	2	2	3300	A
120	3	2	3290	A
180	3	1	3270	A
45	3	3	3250	A
30	2	3	3230	A
60	3	1	3220	A

Table 14. (Continued)

60	3	3	3220	A
60	3	2	3040	A
180	2	3	3000	A
180	3	3	3000	A
180	2	2	2810	A
30	1	3	2650	A
120	3	3	2560	A
30	3	3	2190	B

*A-Well
B-Fair
C-Poor

Table 15. Range of Compressive Strength in Concrete Batch

Mix Time (Seconds)	Replication 1, 2, 3	Range of Compressive Strength Among Positions	Visual Classification of Uniformity A, B, C*
30	1	3200	A
30	3	3200	B
45	3	2920	A
60	1	2350	A
30	2	2190	A
120	1	1290	A
60	2	1280	A
120	3	1190	A
45	2	1150	A
45	1	850	A
180	2	760	A
180	1	750	A
120	2	530	A
180	3	390	A
60	3	180	A

*A-Well Mixed
B-Fair
C-Poor

Table 16. Range of Fineness Modulus in Concrete Batch

Mix Time (Seconds)	Replication 1, 2, 3	Range of Fineness Modulus Among Samples	Visual Classification of Uniformity A, B, C*
180	2	0.99	A
120	3	0.95	A
30	1	0.61	A
45	2	0.47	A
30	3	0.43	B
60	1	0.37	A
60	3	0.32	A
30	2	0.31	A
60	2	0.29	A
45	1	0.27	A
45	3	0.23	A
120	1	0.16	A
180	1	0.13	A
180	3	0.12	A
120	2	0.11	A

*A-Well-Mixed
B-Fair
C-Poor

Table 17. Range of Cement Content within Concrete Batch

Mix Time (Seconds)	Replication 1, 2, 3	Maximum Variation Among Positions (Grams)	Visual Classification A, B, C*
45	3	.22	A
30	3	.14	B
60	1	.13	A
120	3	.12	A
120	1	.09	A
45	2	.08	A
60	3	.08	A
180	1	.07	A
60	2	.07	A
180	2	.07	A
30	2	.06	A
30	1	.05	A
180	3	.05	A
45	1	.04	A
120	2	.04	A

*A-Well-Mixed
B-Fair
C-Poor

CHAPTER V

SUMMARY OF RESULTS AND CONCLUSIONS

Summary of Results.--For the 3.5 cubic-yard batch of Class "A", air-entrained concrete produced in the horizontal-tilting drum type mixer the following results were obtained:

(1) Mix times, replications, and positions used in this experiment contributed significantly to variation in the fineness modulus. None of the interaction terms are significant.

(2) In this experiment, the analysis of variance indicates that the position effect is the sole contributor to variation in compressive cylinder strength.

(3) The position effect contributes significantly to variation in cement content of the mortar samples. Neither the mix time, replications, nor interaction effects cause a significant difference in cement content among samples.

(4) The author was unable to determine visually the degree to which concrete was mixed when correlated with compressive strength, fineness modulus and cement content.

Conclusions.--The ease with which the sampling and testing program described in this thesis can be used in evaluating mixing efficiency justifies its application. Activation analysis appears to be a feasible method for determining the cement content of cast mortar samples. Although not equaling the accuracy obtained by chemical analysis, the

cement content may be predicted with a 95 per cent confidence interval, giving an average error of 11 per cent based upon the point estimate values.

The principal advantages of activation analysis are the ease and speed of cement content determinations. The principal disadvantages are that a laboratory with trained personnel and equipped for irradiating samples and counting their disintegration emissions are required and that the experiment may not be performed on concretes containing aggregate of limestone, marble, or other stone with an appreciable calcium content.

It may be concluded from the data collected that the mixer sampled did not produce a uniform concrete mixture. Regardless of the time mixed, concrete samples from the first third of the discharge stream had a significantly different fineness modulus, compressive strength, and cement concentration than samples drawn from Positions 2 and 3.

Although no conclusions can be drawn about other mixers, the results of this research may be an indication that changes are needed in the blade angles or size of the horizontal-tilting drum design to insure the production of a uniform concrete mixture.

APPENDIX

APPENDIX A

DATA SHEET USED IN PROCESSING AND TESTING OF SAMPLES

Uniformity of Concrete Mix
Date:_____

Type Mixer: Horizontal Tilting Drum, 3.5-Cubic Yds.
Site: MacDougald-Warren Hapeville Plant

Concrete Specifications: Class "A", Air-Entrained
Volume Mixed: 3.5-Cubic Yds.
Mixing Time:_____Seconds

Sample:_____
Replication:_____

Visual Classification
Poor_____Fair_____Well_____

Compressive Strength Test
Cylinder No:_____Testing Date:_____28 Day Failure Load:_____
Compressive Strength:_____Lbs./Sq. Inch

Cement Content by Neutron Activation

Mortar Sample No.	Corrected Sample Count Channels 62-74	Cement Content (Grams) See Fig. 11	Net Sample Weight (Grams)	Cement-Mortar Ratio (Grams Cement Per Gram Mortar)

Sieve Analysis
Total Weight of Sample:_____Grams, Weight of Split:_____Grams

Sieve Size:	Weight Retained	Cumulative Weight Retained	Cumulative Percent Retained
# 1 1/2			
3/4			
3/8			
4			
8			
16			
30			
50			
100			
		Fineness Modulus	
# 1			
1/2			

APPENDIX B

EQUATION-OF LINE CEMENT-CONTENT-VERSUS-COUNT

Table 18. Five-Minute Counts Recorded by Cement Standards

Cement Content Grams	Counts Recorded, Channels 62-74		
	1	2	3
0.6	723	641	702
0.8	959	898	898
1.0	1159	1126	1218
1.2	1310	1379	1378
1.4	1572	1630	1688
1.6	1830	1774	1773
1.8	1967	2042	2073

GENERAL DATA

W represents cement content in grams
$\Sigma Wi = 25.2$; $\overline{W} = \Sigma Wi/n = 1.2$;
$(\Sigma Wi)\,\overline{W} = 30.24$; $\Sigma Wi^2 = 33.60$;
$\Sigma Wi^2 - (\Sigma Wi)\,\overline{W} = \Sigma (Wi - \overline{W})^2 = 3.36$

C represents counts
$\Sigma Ci = 28{,}720$; $\overline{C} = \Sigma Ci/n = 1{,}368$;
$(\Sigma Ci)\,\overline{C} = 39{,}288{,}960$;
$\Sigma Ci^2 = 43{,}456{,}204$;
$\Sigma Ci^2 - (\Sigma Ci)\,\overline{C} = \Sigma (Ci - \overline{C})^2 = 4{,}167{,}244$

$n = 21$; $1/n = 0.0476$

$(\Sigma Wi)\,(\Sigma Ci)/n = \overline{W}\,\Sigma Ci = 34{,}464$;
$\Sigma WiCi = 38{,}196$
$\Sigma WiCi - (\Sigma Wi)\,(\Sigma Ci)/n = \Sigma (Wi - \overline{W})\,(Ci - \overline{C}) = 3732$

Data for Equation of Line:
$b = \Sigma (Wi - \overline{W})\,(Ci - \overline{C})\;\; \Sigma (Wi - \overline{W})^2 = 1111$
$a = C - b\overline{W} = 35$

APPENDIX B (CONTINUED)

Equation of Line = $C = a + bW = 35 + 1111\ W$

$$\text{Correlation Coefficient } r = \frac{\sum_{i=1}^{n}(Wi - \overline{W})\ (Ci - \overline{C})}{\sqrt{\sum_{i=1}^{n}(Wi - \overline{W})^2 \sum_{i=1}^{n}(Ci - \overline{C})^2}}$$

$$= \frac{3732}{\sqrt{3.36 \times 4,167,244}}$$

$$= 0.997$$

95 Per Cent Confidence Limits

$$\frac{C' - a}{b} \pm \frac{t_{\alpha/2,\ n-2} S_{c/w}}{b} \sqrt{1 + \frac{1}{n} + \frac{(\frac{C' - a}{b} - \overline{W})^2}{\sum_{i=1}^{n}(Wi - \overline{W})^2}}$$

$$\frac{C' - 35}{1111} \pm \frac{2.093\ (33.2)}{1111} \sqrt{1 + .0476 + \frac{(\frac{C' - 35}{1111} - 1.2)^2}{3.36}}$$

$C' =$	600,	1000,	1400,	1800
$W' =$	$.508 \pm .068$	$.868 \pm .065$	$1.229 \pm .064$	$1.592 \pm .066$

APPENDIX C

EXAMPLE CALCULATION IN DETERMINING CEMENT CONTENT OF MORTAR SAMPLES

Sample Number	Indium Foil Data				
	Foil Count		True Count N_t	Foil Weight (Mg.)	N_t Per 20 Mg.
	5 Min.	Sec.			
334	77,039	257	299	201	298

Sample Number	Mortar Sample Data						
	Sample Count Channels 62-74	Sample Count Per 300 N_t	Sample Weight (Grams)	Container Weight (Grams)	Net Sample Weight (Grams)	Weight Cement* (Grams)	Cement Content Grams Cement Per Gram Mortar
334	1301	1310	5.18	0.94	4.24	1.14	0.27

*See Figure 11.

APPENDIX D

CRITERIA FOR THE VISUAL GRADATION OF CONCRETE SAMPLES

Well-mixed	Uniform dispersion of batch constituents.
	Proper workability.
Fair	Uniform dispersion of cement and coarse and fine aggregate
	Dry or excessively wet.
Poor	Segregation of one or more constituent of the batch.
	Dry or excessively wet.

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